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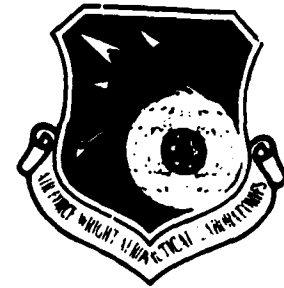
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DETERMINATION OF SELECTED DISTILLATE BLENDING SOLVENTS  
IN SIMPLE AND COMPLEX AIRCRAFT FUEL MATRICES VIA GLASS  
CAPILLARY GAS CHROMATOGRAPHY

Paul C. Hayes, Jr.

Fuels Branch  
Fuels and Lubrication Division

June 1981

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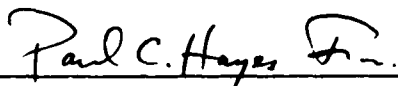
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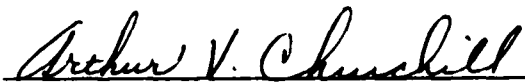
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PAUL C. HAYES, JR.  
Project Scientist, Fuels Branch  
Fuels and Lubrication Division



ARTHUR V. CHURCHILL  
Chief, Fuels Branch  
Fuels and Lubrication Division

FOR THE COMMANDER



ROBERT D. SHERRILL  
Chief  
Fuels and Lubrication Division

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A study was initiated to accurately determine the volume percent of various hydrocarbon distillate solvents present in blends with aircraft jet fuels JP-4 and JP-8. These modified fuels were an integral part of a combustion program to elucidate the effect altered fuel properties had on selected combustion parameters. A method of analysis was desired to check the accuracy of both in-house and contractual blending work. The effort was further expanded to even determine the amount of JP-4 present in the event of gross cross-contamination.		

## 20. ABSTRACT (Cont'd)

Glass Capillary Gas Chromatography was the method of choice. A gas chromatographic process was developed and systematic quantitative analyses performed to substantiate the accuracy and the precision of the new test method. A computing integrator interfaced with the gas chromatograph performed all the data collection and peak area percent calculations automatically, with the results recorded on an internal printer. Least squares curve fitting yielded calibration curves used to determine each blending solvent as well as JP-4 in what proved to be samples of cross-contaminated fuels, e.g. containing both JP-4 and JP-8. A precision statement was also provided at the 95% confidence level.

## FOREWORD

This technical report describes work performed under the In-House work unit 30480591 administered by the Fuels Branch (POSF), Fuels and Lubrication Division (POS), Air Force Wright Aeronautical Laboratories, Aero Propulsion Laboratory (AFWAL/PO). Project scientist for this program was Mr. Paul C. Hayes Jr., who also prepared this report. For the simpler blends analyzed, Mr. Timothy N. Gootee (AFWAL/POFF) operated the capillary gas chromatograph, prepared the calibration standards, and tabulated the raw data. For the complex fuels analyzed, a substantial effort was provided by Mr. Edward W. Pitzer (AFWAL/POFF). He maintained and operated the glass capillary gas chromatographic system throughout this phase of the project. Furthermore, he generated the calibration plots and statistical analyses at a computer terminal, and calculated and tabulated all the final results. Additional assistance was provided by Teresa A. Boos, in crosschecking and retabulating some of the data, and by Kermit R. Redmon, for the initial calibration curve plots.

Particular gratitude is extended to Mr. Edward C. Eimutis, a statistician with Monsanto Research Corporation (Dayton Laboratory), whose stimulating lectures and guidance were invaluable in deriving the precision statements of all of the analyses contained in this report.

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## LIST OF ABBREVIATIONS

ABBREVIATION	MEANING
(GC) <sup>2</sup>	Glass Capillary Gas Chromatography
vol %	percentage of blending solvent by volume
ml	milliliters
n'	effective theoretical plates
RT'	component's adjusted retention time
PW	component's corresponding peak width in time
JP-4	jet propulsion fuel, wide-distillate cut, conforming to MIL-T-5624L
JP-8	jet propulsion fuel, Kerosene distillate cut, conforming to MIL-T-83133A
GMSO	*Gulf Mineral Seal Oil Blending Solvent
2040	*2040 Blending Solvent
Xylene Bottoms	*Xylene Bottoms Blending Solvent

\*Reference: Gleason, C. C., Oller, T. L. Shayeson, M. W., and Bahr, D. W., "Evaluation of Fuel Character Effects on J79 Engine Combustion System," AFAPL-TR-79-2015, CEEDO-TR-79-06, June 1979.

SECTION I  
INTRODUCTION

High resolution glass capillary gas chromatography affords a thoroughly detailed profile of complex hydrocarbon mixtures such as aviation turbine fuels JP-4 and JP-8. JP-4 is a wide-cut distillate jet propulsion fuel conforming to MIL-T-5624, JP-8 a kerosene similar to commercial Jet-A conforming to MIL-T-83133. The experimental study described herein is but another analytical application of the high resolution obtainable with current glass capillary columns.

Specifically, this effort was undertaken to qualitatively identify and quantitatively determine three different hydrocarbon blending solvents used to modify the properties of JP-4 and JP-8. Fuels to be examined were from a contracted combustion system evaluation. The samples included a current JP-4, a current JP-8, and five mixtures each of the JP-4 and the JP-8 with the blending solvents. The modified fuels achieved three different levels of hydrogen content: 12, 13, and about 14 percent by weight. Two different types of aromatics were used to reduce the hydrogen content of the base fuels: a monocyclic aromatic (i.e. xylene bottoms) and a bicyclic aromatic described by the supplier as a naphthalene concentrate (i.e. 2040 solvent). A third blending component used to increase several fuels' final boiling points and the viscosity of two blends was described as a predominately paraffinic, water-white oil (GMSO) (See Reference 1.)

The rationale for the selection of this test fuel matrix was to anticipate future variations in key fuel properties that might be dictated by availability, cost, the change from JP-4 to JP-8 (under consideration since 1968) as the prime USAF aviation turbine fuel, and the use of nonpetroleum sources for jet fuel production.

However, of the two sets of modified fuel samples examined, hitherto called retained and ignition samples, preliminary capillary profiles or chromatograms of the ignition fuels set indicated substantial cross-contamination. In fact, JP-4 was discovered in all ignition fuel samples. Consequently, for calibration purposes, JP-4 and the three blending solvents were considered to be dissolved, in varying amounts, in JP-8. Unknown mixtures would then be analyzed with the aid of the above four calibration curves and the components reported on a volumetric basis in JP-4 fuel.

As stated above, the analytical method of choice was high resolution capillary gas chromatography. A short digression into the meaning and emergence of this technique to the forefront of analytical chemistry is warranted.

Even before Tswett discovered and developed chromatography in 1903, techniques had already been utilized to analyze crude petroleum samples. Shortly before the turn of the century, David Talbot Day, Chief of the Division of Mineral Resources of the American Geological Survey, applied the process of filtration to separate *Pennsylvania petroleum*. His experiments demonstrated that crude oil forced upward through a column of powdered limestone was changed in color and composition (Reference 2). Spanning the 75 years since these humble beginnings, chromatography has established itself as a valuable tool in routine analyses to control refining operations, in research separations and in analytical research.

The remarkable advantages of capillary gas chromatography as applied to the petrochemical industry are speed of analysis, resolution of complex samples into individual components, sensitivity for trace analysis on very small sample volumes, and versatility to glean both qualitative and quantitative information from a wide variety of mixtures. In general, gas chromatography is the physical means of separating a sample into its individual components via distribution of the sample between two phases: a stationary phase (i.e. column's liquid coating) and a mobile phase (i.e. carrier gas). The process requires that the sample be sufficiently volatile to be carried through the column (Reference 3). Owing to the

wide boiling character of conventional hydrocarbon fuels and fuel blends, temperature programming of the column is necessary. Temperature programming is the increase of column temperature with time. Using the proper programming rate, components that elute or emerge early from the column can be well resolved and later emerging peaks made to be sharp and uniformly distributed throughout the chromatogram. When the stationary phase is nonpolar, sample components are separated according to their relative volatility, akin to, but not strictly following, their atmospheric boiling point order.

Glass capillary columns represent a new technology and expanded capabilities for gas chromatographic separations. Essentially what have made (GC)<sup>2</sup> a practical technique are (Reference 4):

- a) commercial availability of both polar and nonpolar capillary columns with guaranteed efficiencies
- b) chemically inert and thermally stable capillary columns and stationary phases
- c) low dead volume detectors, accessories, and instrumentation that preserve the resolution and efficiency of these columns and
- d) more and more capillary chromatography applications appearing in the literature

The efficiency of an exceptionally well-packed conventional 1/8-inch stainless steel column may be the same as for the well-coated glass capillary column used in this study in terms of the number of theoretical plates per meter. However, because the pressure drop across the capillary column is so low, a much longer capillary column can be utilized. Consequently, the total number of theoretical plates in the capillary column may be a factor of 10 to even 100 greater than in conventional 1/8-inch packed columns. Resolving power is proportional to the total number of column plates. This project takes advantage of the vastly increased number of plates available in glass capillary gas chromatography to discern what hydrocarbon components belong to what specific blending solvents dissolved in complex fuel mixtures.

## SECTION II

### EXPERIMENTAL APPROACH

#### 1. INSTRUMENTATION

The basic analytical instrumentation utilized in this study was the varian Model 3700 Capillary Gas Chromatograph equipped with dual-flame ionization detectors. The chromatograph was factory-modified to accept glass capillary columns, and also fitted with a Varian Model 8000 Automatic Liquid Sampler. A Varian Model CDS-111 Computer Integrator controlled the automatic liquid sampler, the temperature programming, and the data collection, and continuously monitored the systems parameters.

The Varian chromatograph was equipped with an all-glass injection and detection system with dead volumes eliminated or reduced to a practical minimum by design and/or carrier purging. The system provided a choice of sampling-injection modes of which the normal split mode was used in this project. The capillary pneumatics was a pressure regulated system, with all the active components thermostatted to better than  $\pm 1^\circ\text{C}$  for maximum retention time repeatability and detector baseline stability. The injector-splitter featured a true positive septum purge which effectively eliminated septum bleed (Reference 5).

#### 2. CAPILLARY GAS CHROMATOGRAPHIC CONDITIONS

The glass capillary column used in this work was manufactured by Scientific Glass Engineering, Inc. (S.G.E. of Austin, Texas) to the following specifications: column length of 62.3 meters, column internal diameter of 0.25 mm, and stationary liquid phase of relatively nonpolar SP2100. The flow rate of the nitrogen carrier gas (1.0 ml/min) was boosted to 35 ml/min at the detector, via make-up nitrogen, to sharpen the eluting peaks. The nitrogen was "high purity" water-pumped, and was further purified via a train of four Matheson Gas Products Gas Purifiers, Model 450, to remove any gross contamination inadvertently introduced, i.e., oil, water, particulate matter ( $\phi 5$  microns). To preserve the integrity of the glass capillary column, a General Electric Model "Go-Getter" was installed downstream from the above four traps to remove residual oxygen or water vapor present to less than 0.1 parts-per-million.

Other (GC)<sup>2</sup> operating parameters were as follows:

- a) Temperature program was from 45°C to 270°C at 5 C°/min., with an upper temperature hold of 15 minutes
- b) Flow rates of hydrogen and compressed air to the detector were 30 ml/min and 300 ml/min, respectively
- c) Injection port and detector were maintained at 300°C;
- d) Electrometer was set at range,  $10^{-11}$  and attenuation, 1
- e) Injection volume was fixed at one microliter
- f) Split ratio set at 85:1
- g) The column was conditioned prior to usage to remove volatile impurities at 280°C overnight with a carrier pressure of 5 psig
- h) Carrier pressure was adjusted to 26 psig
- i) Under the above operating conditions and given the temperature environment of the laboratory, a sample was injected every 70 minutes.

The column efficiency was determined at the factory to be over 175,000 effective plates, which is representative of a moderately efficient column for that length. The sample (S.G.E. Test Kit #5) used for efficiency measurements was a 0.5 microliter injection of a 1% hexane solution of normal alkanes run isothermally at 220°C with a 150:1 split ratio and at a carrier pressure of 26 psig. The peak selected to measure was C<sub>22</sub>. The equation used for calculating the column's effective plates (n') was:

$$n' = 16.0 \left( \frac{RT'}{PW} \right)^2$$

where: a) RT' = the adjusted retention time of C<sub>22</sub>, i.e. its absolute retention time minus the column dead volume time (for unretained normal butane);

b) PW = peak width, i.e. the portion of the peak's baseline intersected by tangents drawn to the peak's sides (time units).

### SECTION III

#### GENERAL METHODOLOGY FOR QUANTITATION

##### 1. BACKGROUND

The method used to relate processed data from the flame detector's electrometer, i.e. area counts, to the composition of the sample was peak area normalization without response factors. In area normalization, the percentage of peak "X" is given by the formula below, where the area of peak "X" is divided by the total area of all the peaks detected in the chromatogram.

$$\% X = \frac{\text{AREA X}}{\text{SUMMATION ALL PEAK AREAS}} \times 100\%$$

Several assumptions have been taken in utilizing this technique:

- a) All of the injected sample is eluted.
- b) All of the components are detected.
- c) The difference in response of the detector for different types of hydrocarbons was not critical or was accounted for.

The nature of the samples investigated, i.e. blending solvents and base fuel, were all hydrocarbon distillates of a distinct boiling point range. It was highly unlikely that there would be polymeric or high molecular weight hydrocarbon material present that could be held up on the column and never eluted. Furthermore, those components to which the flame ionization detector does not respond, i.e. the light fixed gases, air, or water, should be only negligible detractors to the method's quantitative accuracy, because of the restricted nature of the samples studied.

Figures 1 through 4 are capillary chromatograms of the kerosene distillate JP-8, and each of the hydrocarbon blending solvents used as fuel property modifiers. Under the chromatographic conditions employed, JP-4 exhibited over 270 peaks, JP-8 over 245 peaks, and the pure blending solvents had over 185 peaks each. However, nearly all of the individual

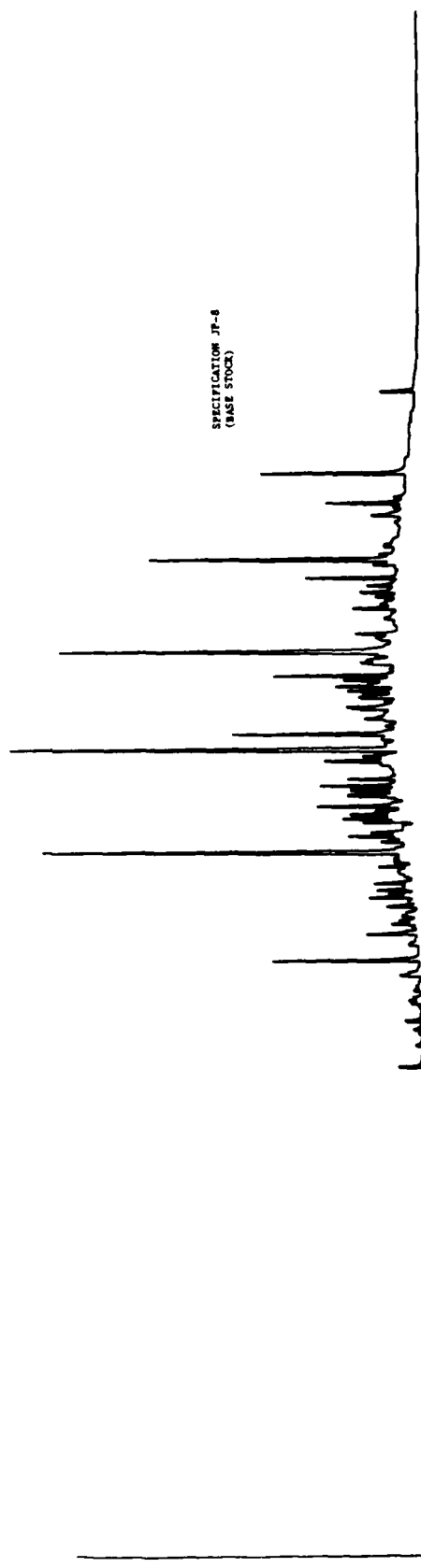


Figure 1. Specification JP-8 (Base Stock)

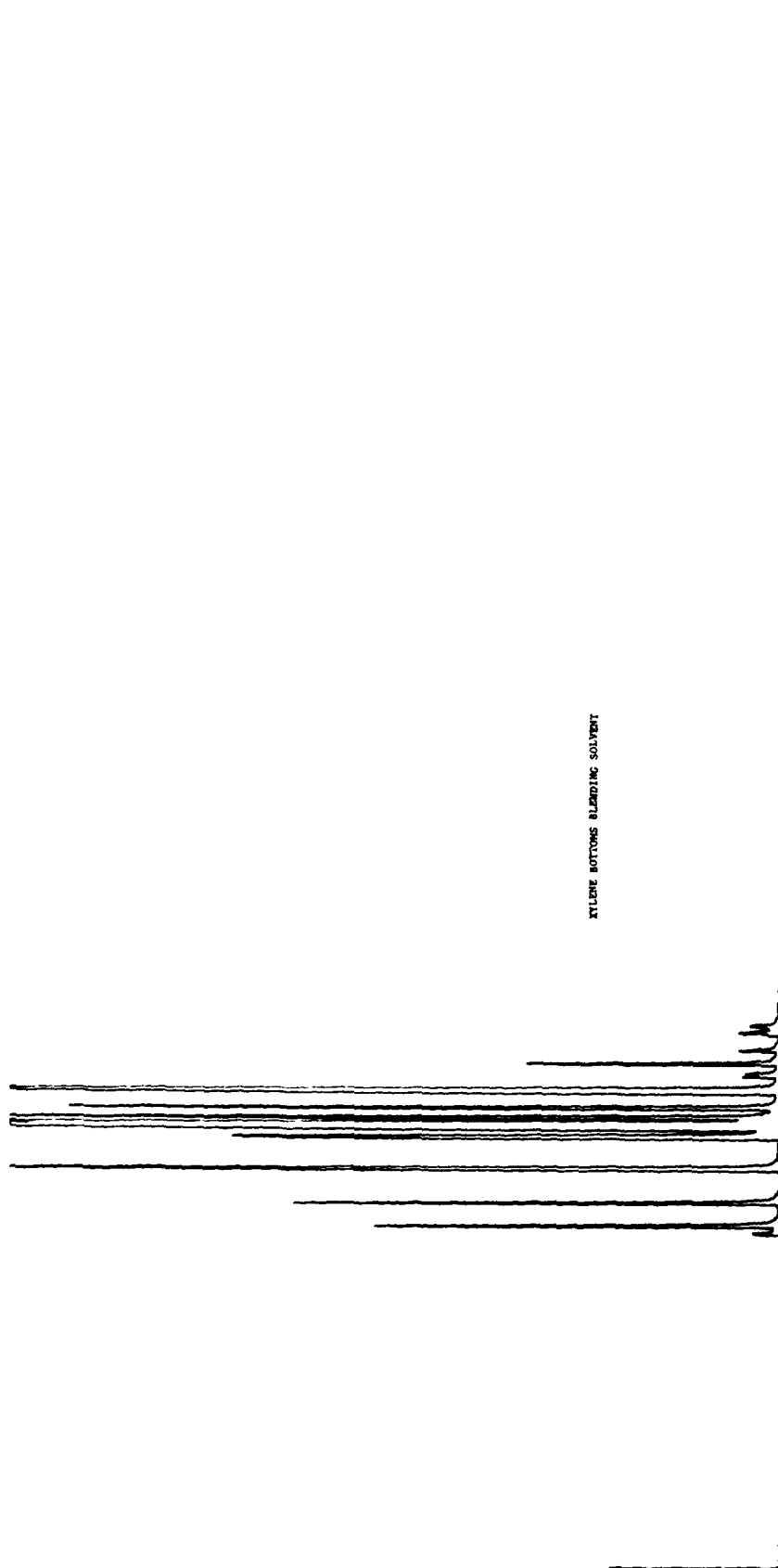


Figure 2. Xylene Bottoms Blending Solvent

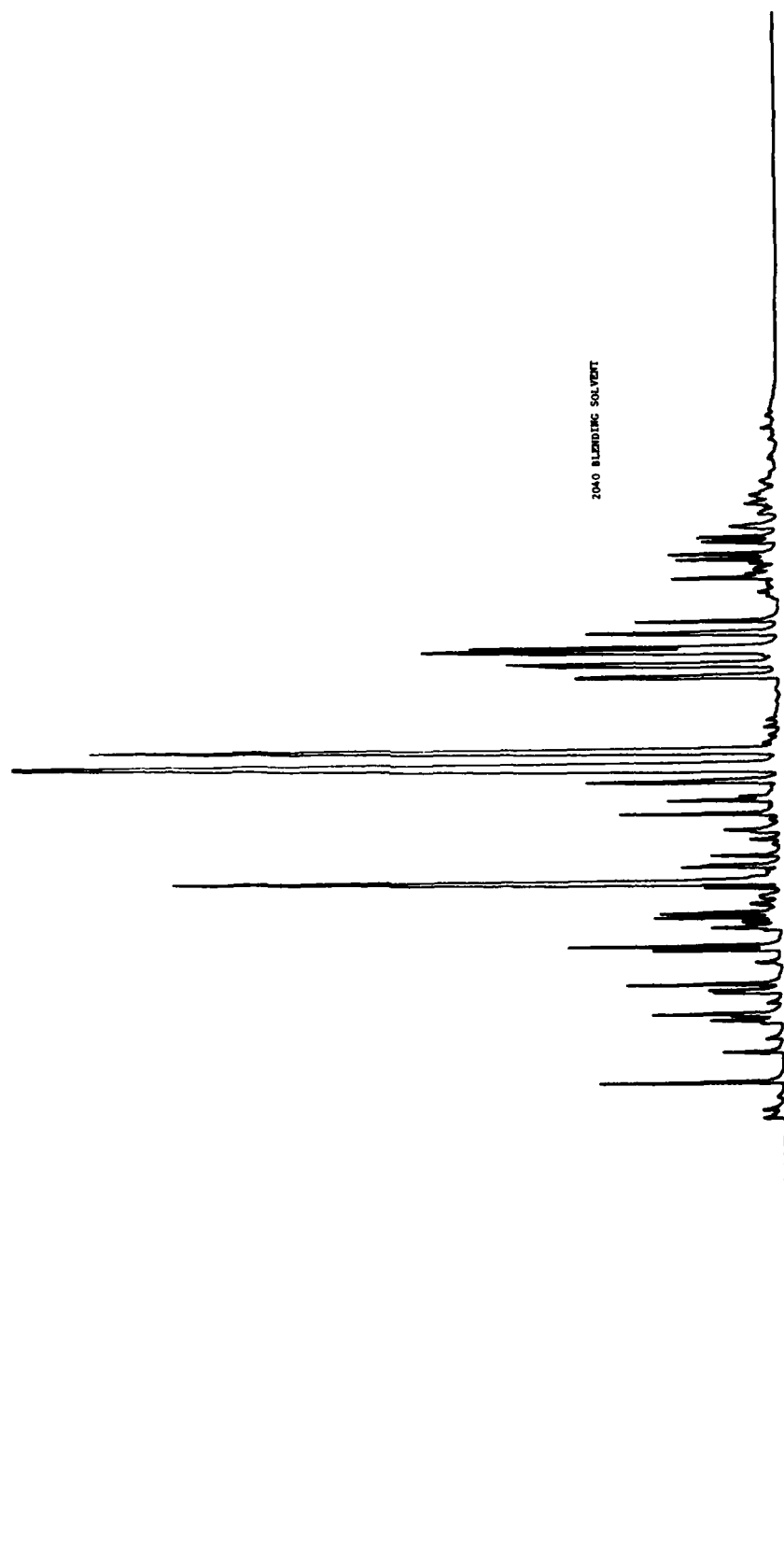


Figure 3. 2040 Blending Solvent

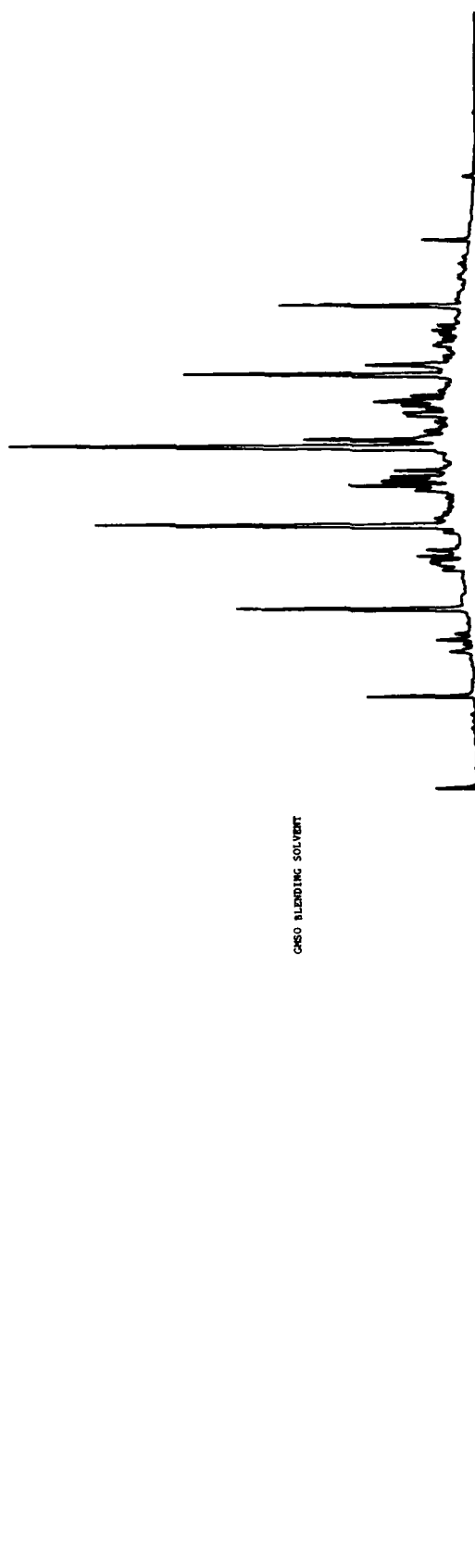


Figure 4. GMSO Blending Solvent

individual hydrocarbons of each blending solvent were also present in both JP-4 and JP-8.

## 2. SIMPLE BLENDS IN JP-8

### a. Preliminary Work

To differentiate solvent from fuel in the unknown samples, each solvent's relatively narrow distillation range was exploited. Chromatographic runs were made on each of the pure blending solvents, as well as on pure JP-8. Each integrator printout, containing hydrocarbon retention times and corresponding area percentages, was arranged side-by-side with the others. A retention time window was judiciously selected for each blending solvent. Each unique window enclosed a substantial area percentage of one solvent but as little cross interference as possible from the other blending solvents and JP-8.

For example, xylene bottoms' time window applied to a sample of 100% xylene bottoms accounted for over 73 percent (by area) of that solvent. On the other hand, when the xylene window was applied to a chromatographic run of pure JP-8, an interference of only 4 area percent was found. As for pure 2040 solvent and pure GMSO, only about 2 and 0 area percent, respectively, appeared in the xylene bottoms' window.

Table 1 has the resulting area percentages for each unique window applied to all of the pure blending solvents and JP-8. This table set the groundwork for correlating chromatographic area percentages with blended volume percentages. For example, if an unknown sample were chromatographed and the peak area percentage found in the xylene bottoms' window was slightly over 73, the sample was pure, i.e., 100% xylene bottoms by volume. However, if an unknown gave a peak area percentage for the same window of only 4, the xylene bottoms' volume percentage was 0.

TABLE 1. PEAK AREA PERCENTAGES FOUND IN EACH UNIQUE RETENTION TIME WINDOW

BLENDING STOCK TIME WINDOW	CORRESPONDING PEAK AREA PERCENTAGES (%)			
	PURE SYLENE BOTTOMS	PURE 2040 SOLVENT	PURE GMSO SOLVENT	PURE JP-8 STOCK
Xylene Bottoms	73.85	1.83	0.00	3.92
2040 Solvent	0.00	54.89	0.00	31.12
GMSO Solvent	0.58	1.95	65.86	3.94

## b. Calibration Blends

For each blending stock, a series of volumetrically accurate calibration blends was prepared at concentrations ranging from 0 to 100% in JP-8. All the blends were chromatographed and the appropriate window area percentages tabulated with the corresponding known volume percentages. These results were then plotted to yield calibration curves specific for each blending solvent (see Figures 5 through 7). Note that the plots have nonzero intercepts. Statistical analysis of this data by least squares curve fitting gave correlation coefficients that in all cases approaches unity (see Table 2). With the linear equation of each unique calibration curve, unknown fuel samples could be quantitatively analyzed for any blending component. A chromatographic run's area percentage, from the proper retention windows was substituted into the formula and the equation solved for volume percentage.

TABLE 2

## STATISTICAL PARAMETERS OF CALIBRATION CURVE PLOTS FOR BLENDING

## SOLVENTS IN BASE JP-8

<u>BLENDING SOLVENT</u>	<u>CORRELATION COEFFICIENT</u>	<u>STANDARD ERROR OF ESTIMATE</u>	<u>SLOPE OF LINE</u>	<u>INTERCEPT OF LINE</u>	<u>EQUATION OF LINE*</u>
Xylene Bottoms	.9995688	.8959970	1.4231	-6.4235	$y = 1.4231 x - 6.4235$
2040 Solvent	.9975142	2.282028	4.2223	-134.33	$y = 4.2223 x - 134.3359$
QMSO Solvent	.9996427	.8744594	1.6050	-5.1400	$y = 1.6050 x - 5.1400$

Note:  $y = (CC)^2$  Peak Area Ratio  
 $x =$  Known Volume Percent

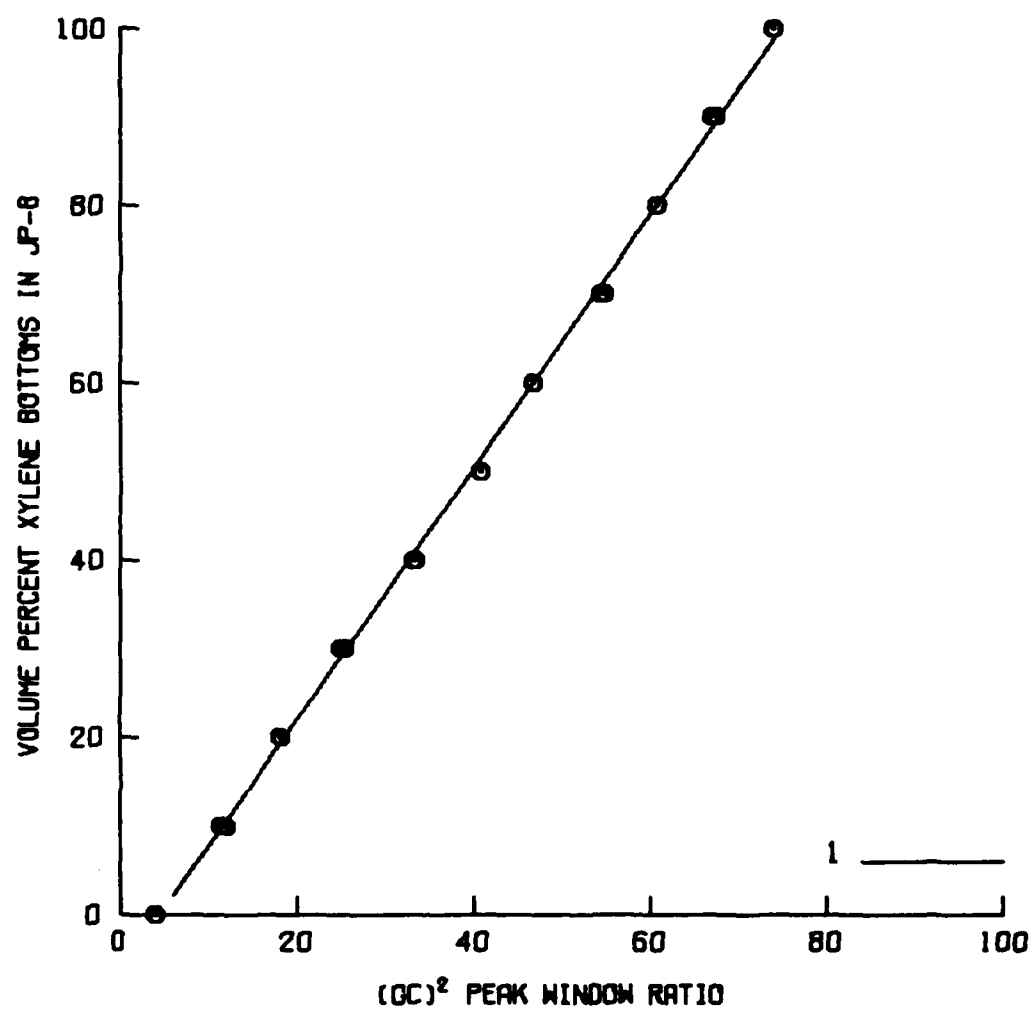


Figure 5. Calibration Curve for Xylene Bottoms in JP-8 By  $(GC)^2$

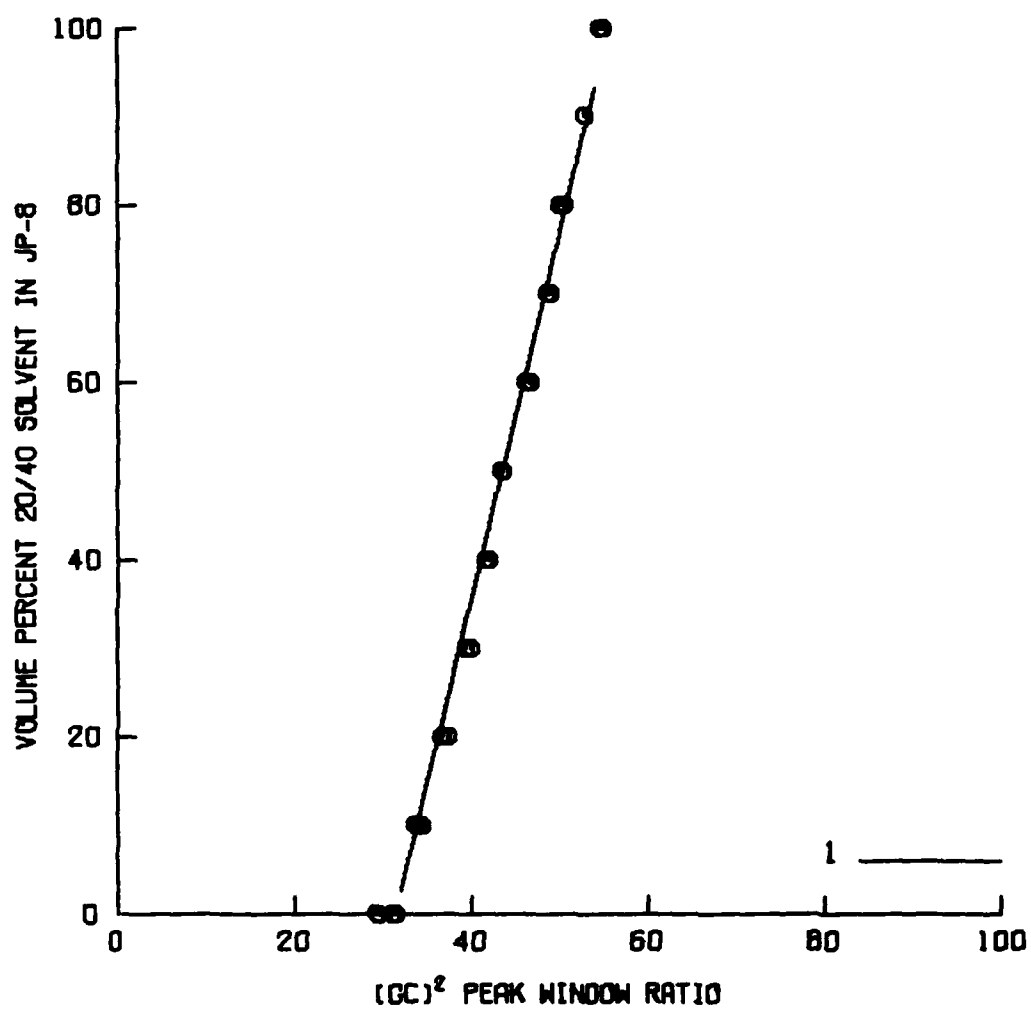


Figure 6. Calibration Curve for 2040 Solvent in JP-8 By  $(GC)^2$

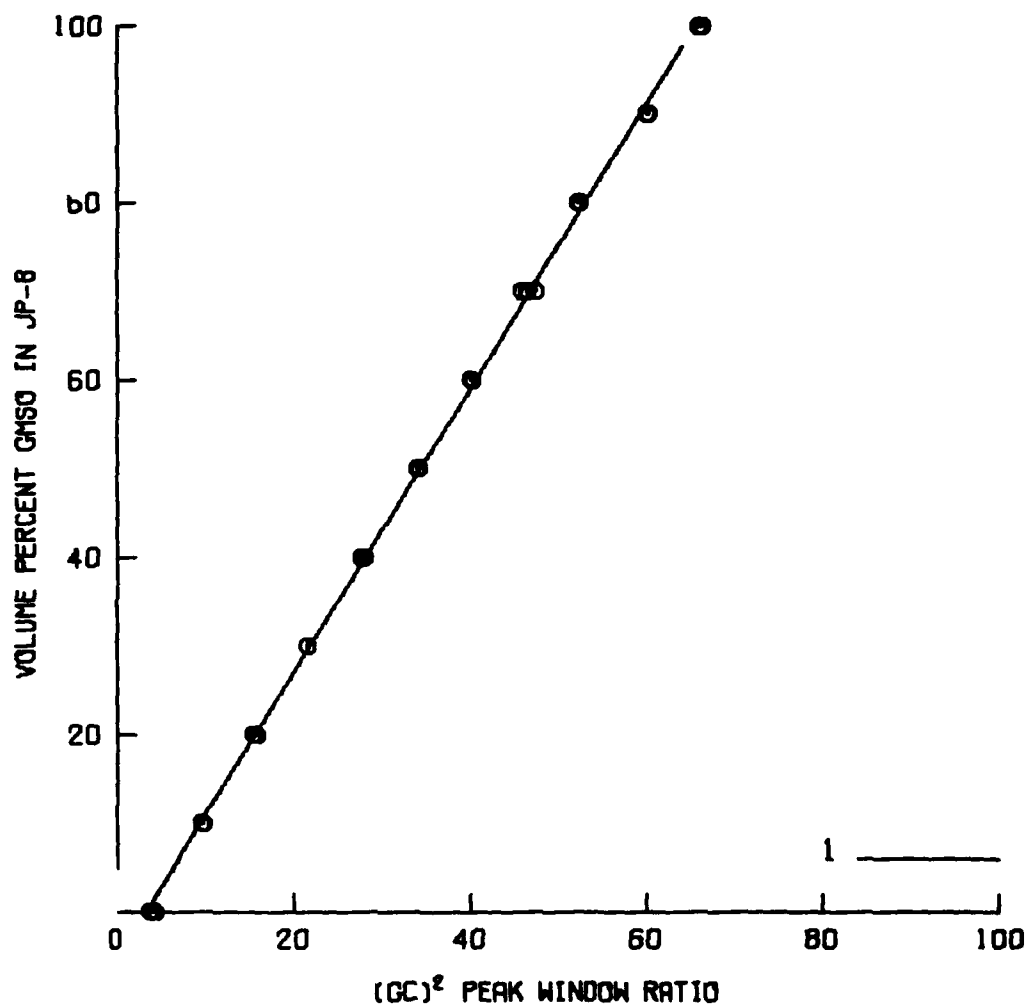


Figure 7. Calibration Curve for GMSO in JP-8 by (GC)<sup>2</sup>

The equation of the linear least squares line for each blending stock is given by the appropriate slope and intercept in Table 2. These equations were then rearranged so that the amount of any of the three blending solvents in an unknown sample, i.e. "x", could be predicted given the appropriate area percentage from a (GC)<sup>2</sup> run, i.e. "y". The standard error of estimate cited above has properties analogous to those of the standard deviation about a mean. Lines constructed parallel to each regression line, of x values (volume % of blending solvent) for given y values (peak area % in blending solvent window), at respective vertical distances of one, two and three standard deviations from it, include about 68%, 95%, and 99.7% of the data points, respectively (Reference 4). Small errors of estimate mean precise, repeatable results.

Note that the poorest error of estimate was for the case of the 2040 solvent where the retention time window unfortunately had to include considerable, i.e., 31% of the base JP-8. Thus, there was not as great a relative area difference in 2040 blends ranging from 0 to 100%. The resulting regression line has a steep slope where a few peak area percentages meant several blended volume percentages.

#### c. Back Analysis for Accuracy Estimation Statement

The anticipated range of amounts of each blending solvent in JP-8 for the combustion effort is included in Table 3. Also displayed are the results of back-analyses of the corresponding calibration blends utilizing the equations of the least-square regression lines.

TABLE 3. BACK-ANALYSIS OF ANTICIPATED BLENDS FOR EACH SOLVENT IN JP-8

BLENDING SOLVENT	KNOWN (ANTICIPATED) VOLUME %	BACK-CALCULATED VOLUME %	KNOWN (ANTICIPATED) VOLUME %	BACK-CALCULATED VOLUME %
XYLENE BOTTOMS	50.00	51.48	20.00	19.22
2040 SOLVENT	40.00	42.45	10.00	8.85
CMISO SOLVENT	20.00	20.12	10.00	10.40

As expected, the poorest accuracy occurs with the determination of 2040 solvent in JP-8, the one blending solvent with the standard error of estimate greater than 1.00.

### 3. COMPLEXLY BLENDED FUELS

#### a. Preliminary Work

As in the case of the previous solvent blends, in JP-8 a unique time window was also selected for JP-4 from a chromatographic run (see Figure 8). However, since JP-4 is a wide-boiling distillate cut, many of its hydrocarbon components overlapped into the previously selected solvent windows and an entirely new set of time windows had to be generated.

Now, when xylene bottoms' window was applied to a sample of pure xylene bottoms, it accounted for over 82 percent (by area) of that solvent. On the other hand, the xylene bottoms' window applied to a chromatographic run of pure JP-4 gave only about 6.5 area percent. As for pure 2040 solvent, pure GMSO, and pure JP-8, only about 2.3, 0.0, and 2.9 area percentages, respectively, appeared in the xylene bottoms' window.

Table 4 has the resulting area percentages for each unique window applied to all of the pure blending solvents including JP-4 and JP-8. This table set the groundwork for correlating chromatographic area percentages with blended volume percentages for each solvent and JP-4. If an unknown sample was chromatographed and the peak area percentage found in the xylene bottoms' window was slightly over 82, the sample was reported as pure, i.e. 100% by volume, xylene bottoms.

However, selection of the 0% by volume reference, i.e. no blending solvent present, was not straightforward for the ignition fuel samples, since both JP-4 and JP-8 were present. Unfortunately, for a given time window the corresponding background interferences from each base fuel were different (see Table 4). The background interference occurring in each retention time window was subsequently defined in terms of an equi-volume mixture of JP-4 and JP-8, i.e. 50/50 JP-4:JP-8. Such a complex reference background (see Table 4) was realistic for a 0% by volume point,

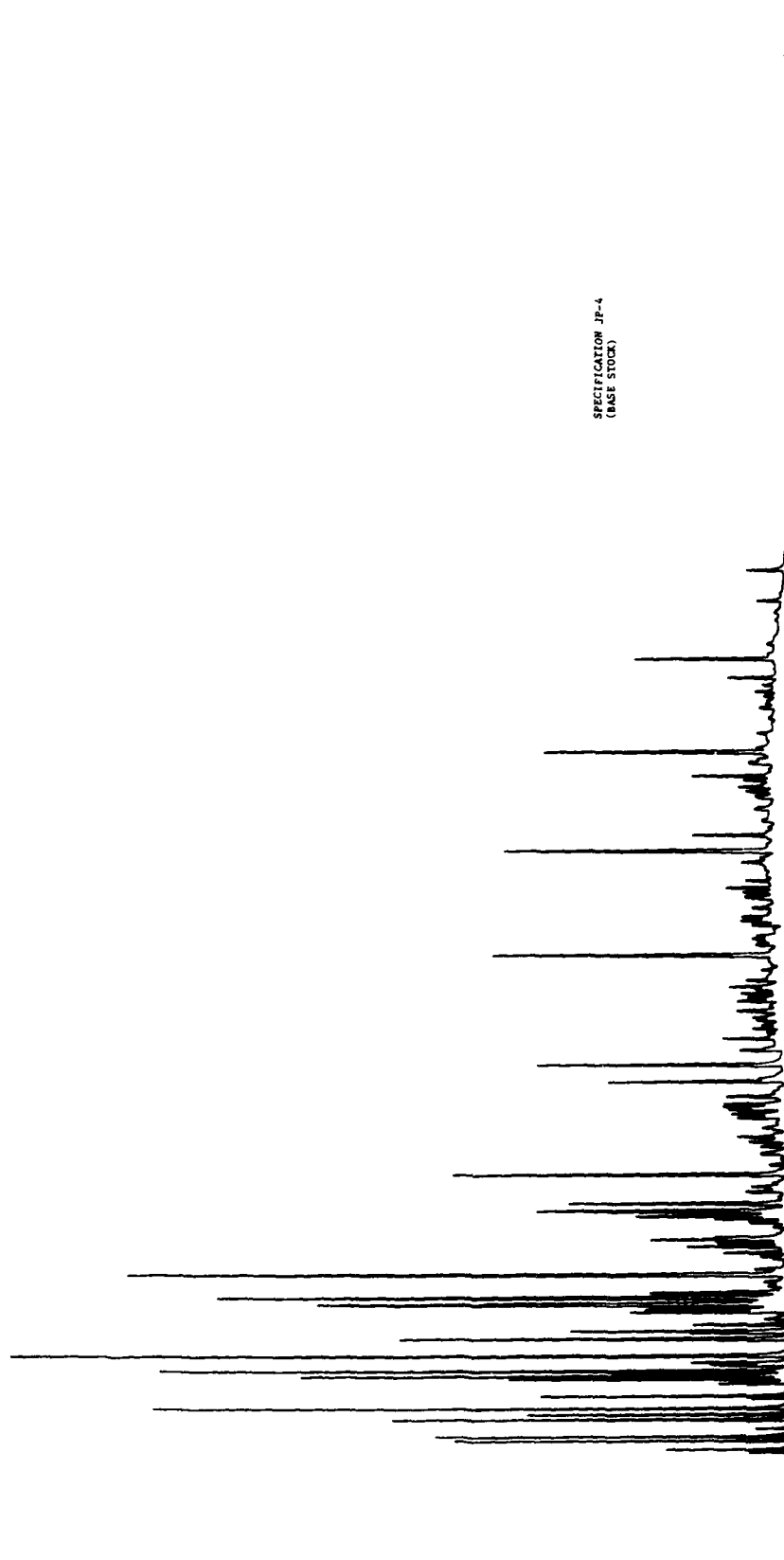


Figure 8. Specification JP-4 (Base Stock)

TABLE 4

PEAK AREA PERCENTAGES FOUND IN EACH UNIQUE RETENTION TIME WINDOW

Blending Stock Time Window	CORRESPONDING PEAK AREA PERCENTAGES (%)					
	Pure JP-4	Pure Xylene Bottoms	Pure 2040 Solvent	Pure GMSO	Pure JP-8	50/50 JP-4: JP-8 Mix
JP-4	39.14	0.00	0.11	0.00	0.49	N/A
XYLENE BOTTOMS	6.52	82.65	2.33	0.00	2.95	4.46
2040 SOLVENT	0.76	0.00	9.12	0.00	1.02	1.04
GMSO	0.44	0.58	1.77	49.03	0.29	0.20

N/A = Not applicable for this blending solvent

representing gross cross-contamination in the ignition fuels. Compared to the simple fuel blends, the entire analytical scheme for the complex blends was geared primarily to examine samples characterized as JP-4/JP-8 mixtures.

Of necessity, this approach also requires a unique time window for JP-4. Pure JP-4's early eluting peaks did not, for the most part, appear in the chromatogram of pure JP-8, nor in any of the blending solvents. This portion of the chromatogram gave the retention time window specific for JP-4. However, the 0% by volume point for JP-4, i.e. no JP-4 present, could not be based on a 50/50 JP-4:JP-8 reference as had all the blending solvents. The 0% by volume point was referenced to the corresponding interference from pure JP-8. For example, the JP-4 retention time window accounts for over 39 area percentage of pure JP-4, i.e. representing 100% by volume. The JP-4 window includes only minimal background interference when applied to pure JP-8, i.e. denoting 0% by volume JP-4 (see Table 4). Note that the area percentage overlap from one blending solvent into another's unique window was relatively small in all cases.

#### b. Calibration Blends

A series of volumetrically accurate calibration blends was prepared. These synthetic blends were complex, in anticipation of complicated ignition fuel compositions. Each calibration standard included each blending solvent and JP-4 in different concentrations in JP-8. The concentration ranges of each solvent and JP-4 were distinct and bracketed the levels expected in the cross-contaminated ignition fuel samples.

All the calibration blends were chromatographed and the appropriate window area percentages tabulated with the corresponding known volume percentages. These results were plotted, incorporating the above 0% and 100% by volume points. Each blending solvent and JP-4 had a specific calibration curve as displayed in Figures 9 through 12.

Note that the plots have nonzero intercepts, reflecting the minimal but nonnegligible background interferences of the 0% point references, i.e. JP-8 and the 50/50 mix.

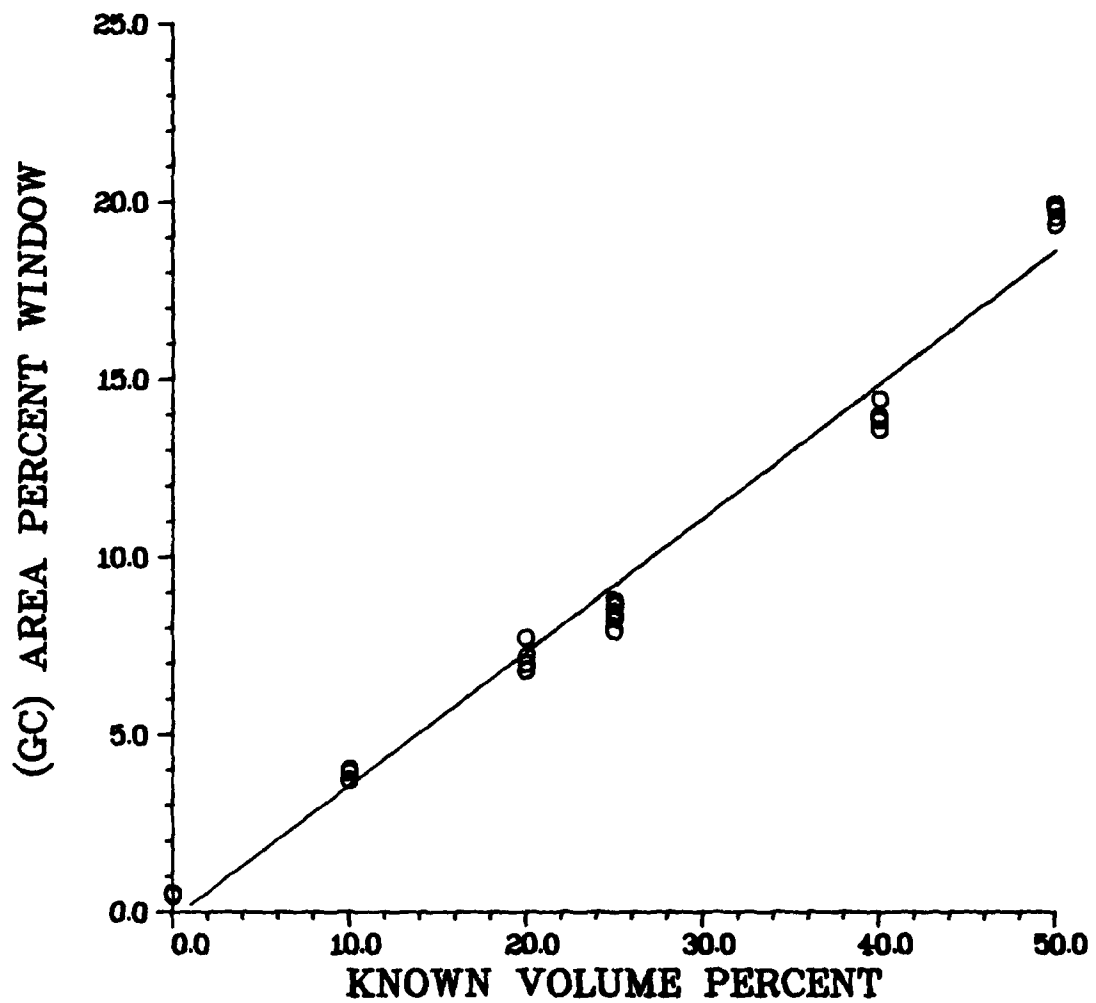


Figure 9. Calibration Curve for JP-4

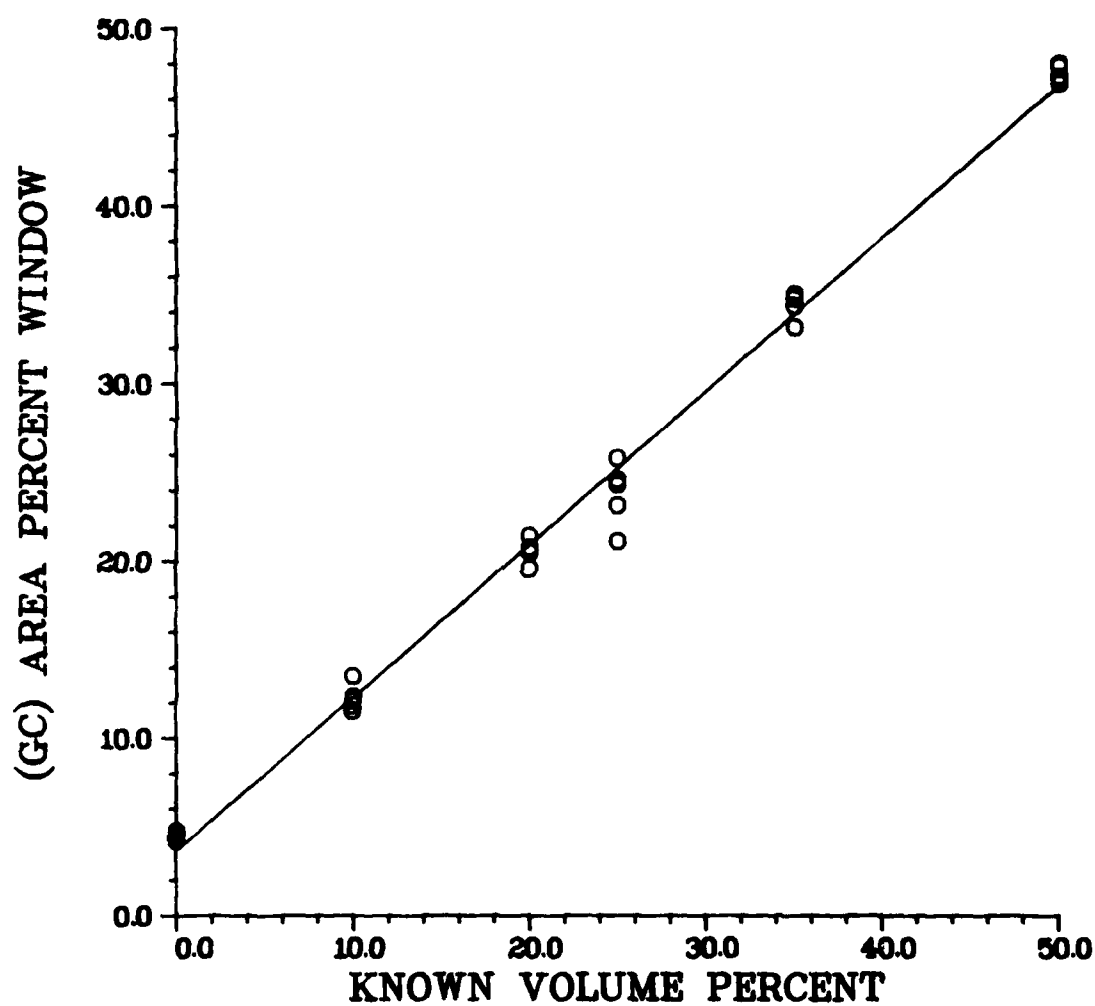


Figure 10. Calibration Curve for Xylene Bottoms

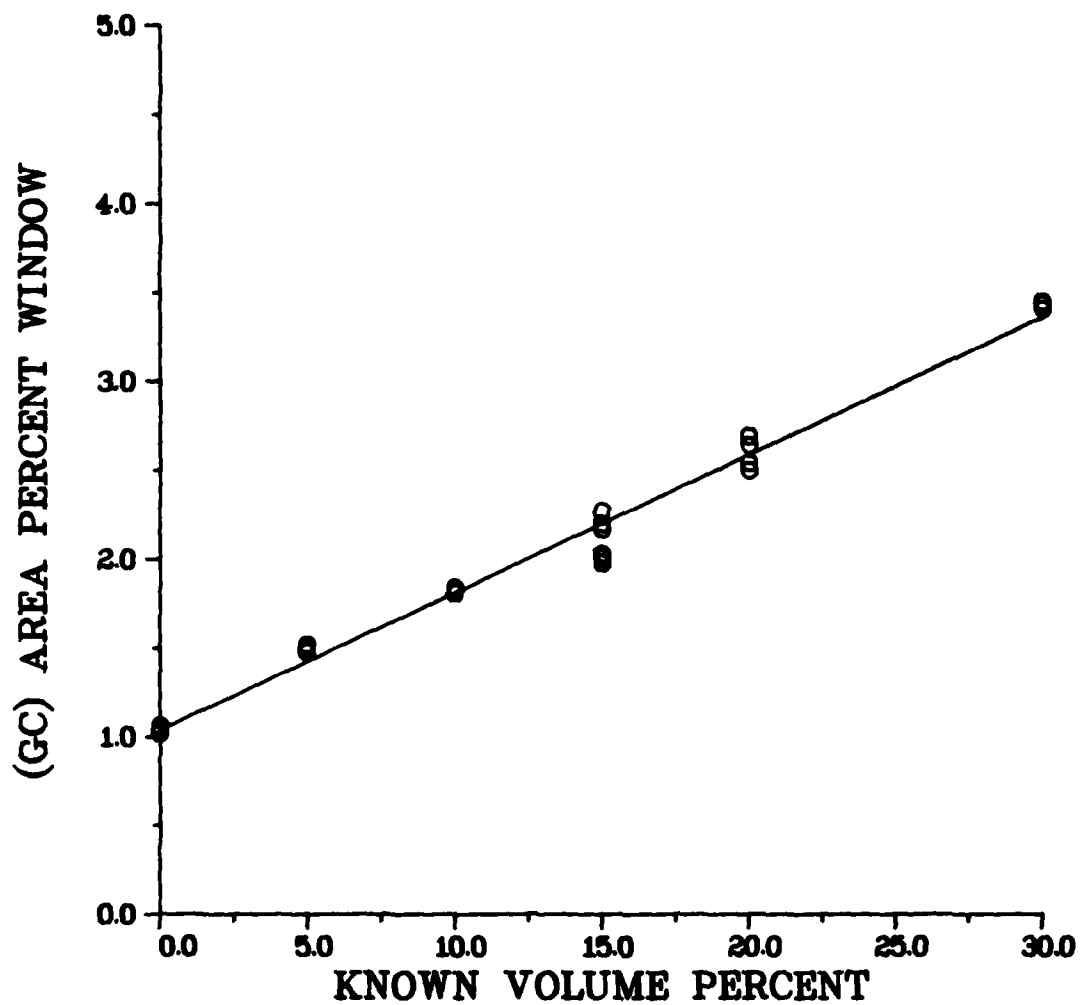


Figure 11. Calibration Curve for 2040 Solvent

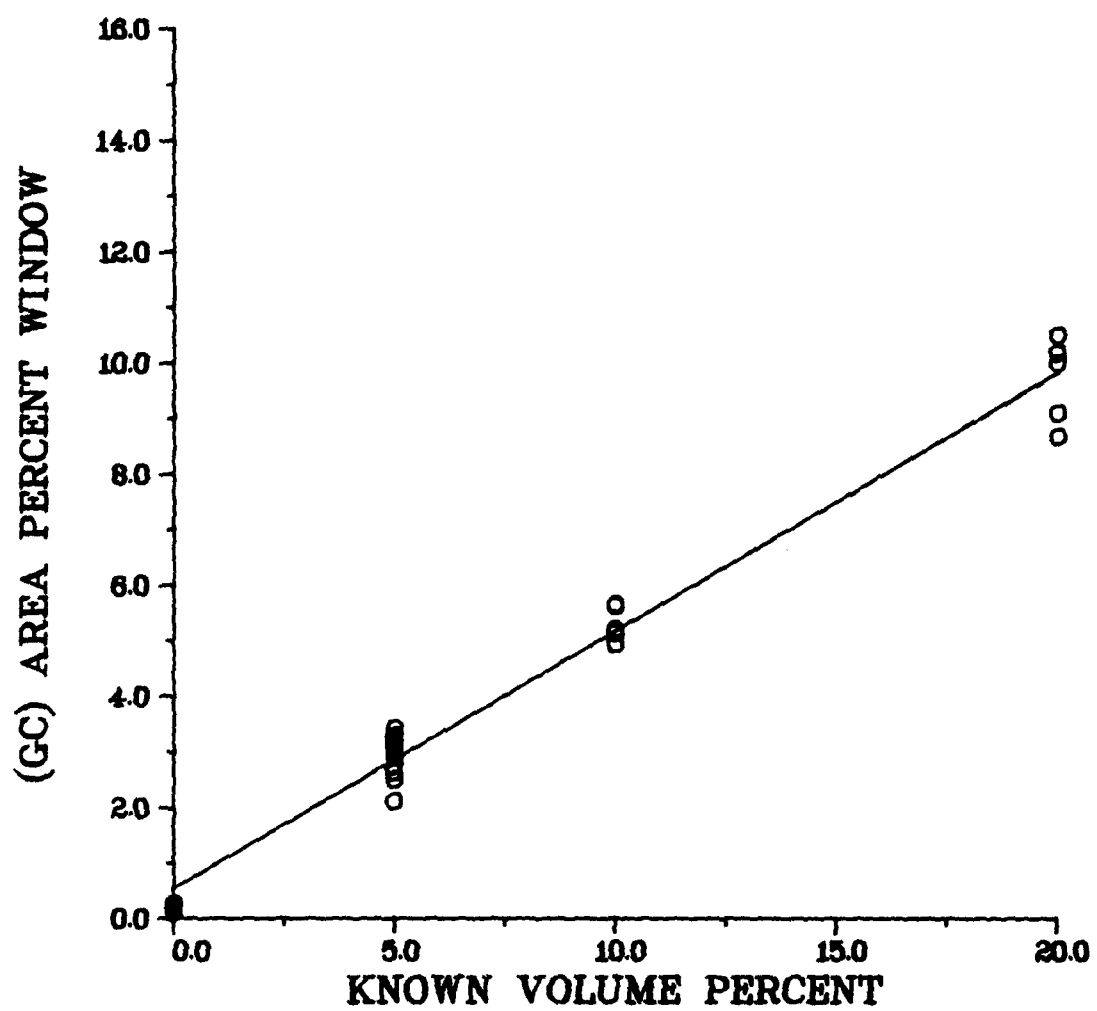


Figure 12. Calibration Curve for GMSO

Statistical analysis of the data via least squares curve fitting yielded correlation coefficients that in all cases approached unity. Table 5 gives some of the statistical parameters of each blending solvent and JP-4's calibration curve.

TABLE 5  
STATISTICAL PARAMETERS OF CALIBRATION CURVE PLOTS  
FOR JP-4/BLENDING SOLVENTS

<u>BLENDING STOCK</u>	<u>CORRELATION COEFFICIENT</u>	<u>LINE SLOPE</u>	<u>LINE INTERCEPT</u>	<u>EQUATION OF LINE*</u>
JP-4	0.994175	0.3748	-0.1476	$y = 0.3748 x - 0.1476$
xylene bottoms	0.996263	0.8614	3.7211	$y = 0.8614 x + 3.7211$
2040 solvent	0.991209	0.0766	1.0589	$y = 0.766 x + 1.0589$
GMSO	0.988717	0.4632	0.5656	$y = 0.4632 x + 0.5656$

\*Note:  $y = (GC)^2$  Peak Area Ratio  
 $x =$  Known Volume Percent

The equation of the linear least squares regression line for each blending solvent and JP-4 is given by the appropriate slope and intercept in Table 5. Each sample line's equation was rearranged, solving for volume percent. A chromatographic run on an unknown sample yielded area percentages for each corresponding time window which when substituted into the proper component's equation would give predicted volume percentages.

#### c. Accuracy and Precision Statements

Two randomly selected calibration standards exemplify both the complexity of these blends and the accuracy of back-calculated results utilizing the equations of the least squares regression lines. Table 6 also includes a precision statement at the 95% confidence level for each back-calculated result. Note that the true volume percentages of the components in the known blends were subject to some minor pipetting errors.

TABLE 6

## BACK-ANALYSES OF COMPLEX BLENDS CONTAINING JP-4 AND BLENDING SOLVENTS IN JP-8

CALIBRATION STANDARD CODE	KNOWN VOL % JP-4	FOUND VOL % JP-4	KNOWN VOL % XYL P.	FOUND VOL % XYL B.	KNOWN VOL % 2040	FOUND VOL % 2040	KNOWN VOL % GMSO	FOUND VOL % GMSO
#5	50.00 ±0.05	50.82 ±2.58	10.00 ±0.02	11.46 ±1.52	10.00 ±1.34	9.94 ±1.34	10.00 ±0.02	9.66 ±1.05
#20	20.00 ±0.03	19.15 ±1.99	20.00 ±0.03	19.03 ±1.38	20.00 ±0.03	19.86 ±1.32	20.00 ±0.03	20.43 ±1.26

The precision statements listed above mean that there is a 95% probability that the true volume percent for a given determination lies within +/- the confidence limits in Tables 6 through 8. They were provided and explained by Mr. Edward Eimutis, a statistician from Monsanto Research Corporation (Dayton Laboratory). The definitions of the statistical parameters and all pertinent references are included in the Appendix.

## d. Unknown Complex Fuels Analyses

Tables 7 and 8 list the calculated volume percentages of JP-4 and blending solvents in the two sets of fuel samples, (retained and ignition) with their corresponding 95% confidence limits. Also included are the originally desired concentrations of each of the above components for each fuel.

Several comments should be made in the interpretation of the results on the unknown fuel samples displayed in Tables 7 and 8. As previously stated, the calibration curves were designed to reduce the error in the analysis of complex solutions containing both JP-4 and JP-8. It should not be surprising that the analyses of the retained fuels set (see Table 7) which were not cross-contaminated would show some minor deviations from the proposed component concentrations, e.g. 104.82% vs 100% JP-4 in DDA/JP-4/78/C2006, 3.32% vs 0% xylene bottoms in the same sample, and the apparent trace amount of JP-4 in the last six fuels (though ignorable according to the respective confidence limits). However, the difference

TABLE 7 ANALYSES OF JP-4/BLENDING SOLVENTS IN CONTRACTED COMBUSTION FUELS:  
"RETAINED" FUEL SAMPLES

"RETAINED" FUEL IDENTIFIER	JP-4 VOLUME %		XYLENE BOTTOMS VOL %		2040 SOLVENT		VOL %		GMSO SOLVENT VOL %	
	PROPOSED	FOUND	PROPOSED	FOUND	PROPOSED	FOUND	FOUND	FOUND	PROPOSED	FOUND
DDA/JP-4/78/C2006	100	104.82 $\pm 4.10$	0	3.32 $\pm 1.75$	0		<1 $\pm 1.71$		0	<1 $\pm 1.26$
DDA/4A2/78/C2006	65	61.73 $\pm 2.49$	0	1.21 $\pm 1.52$	35		45.97 $\pm 2.28$		0	<1 $\pm 1.19$
DDA/4A3/78/C2006	79	81.90 $\pm 3.09$	0	1.86 $\pm 1.51$	21		24.95 $\pm 1.45$		0	<1 $\pm 1.27$
DDA/4X2/78/C2006	48	43.00 $\pm 2.10$	52	54.97 $\pm 1.80$	0		<1 $\pm 1.70$		0	<1 $\pm 1.27$
DDA/4X3/78/C2006	70	70.38 $\pm 2.73$	30	33.64 $\pm 1.45$	0		<1 $\pm 1.62$		0	<1 $\pm 1.11$
DDA/4XG/78/C2006	79	81.63 $\pm 3.08$	9	12.05 $\pm 1.41$	0		<1 $\pm 1.59$		12	13.09 $\pm 1.20$
DDA/JP-8/78/C2006	0	1.70 $\pm 2.56$	0	<1 $\pm 1.79$	0		<1 $\pm 1.61$		0	<1 $\pm 1.27$
DDA/8A2/78/C2006	0	2.07 $\pm 2.21$	0	<1 $\pm 1.66$	31		42.70 $\pm 2.13$		0	<1 $\pm 1.26$
DDA/8A3/78/C2006	0	1.78 $\pm 2.36$	0	<1 $\pm 1.80$	15		23.12 $\pm 1.49$		0	<1 $\pm 1.27$
DDA/8X2/78/C2006	0	1.75 $\pm 2.21$	48	46.27 $\pm 1.63$	0		<1 $\pm 1.62$		0	<1 $\pm 1.11$
DDA/8X3/78/C2006	0	1.30 $\pm 2.56$	24	24.82 $\pm 1.89$	0		<1 $\pm 1.71$		0	<1 $\pm 1.27$
DDA/8GX/78/C2006	0	1.59 $\pm 2.36$	0	<1 $\pm 1.65$	0		<1 $\pm 1.55$		12	10.87 $\pm 1.06$

TABLE 8 ANALYSES OF JP-4/BLENDING SOLVENTS IN CONTRACTED COMBUSTION FUELS:  
"IGNITION" FUEL SAMPLES

"IGNITION" FUEL IDENTIFIER	JP-4 VOLUME %		XYLENE BOTTOMS VOL %		2040 SOLVENT		VOL %		GMSO SOLVENT		VOL %	
	PROPOSED	FOUND	PROPOSED	FOUND	PROPOSED	FOUND	PROPOSED	FOUND	PROPOSED	FOUND	PROPOSED	FOUND
DDA/JP-4/78/C2006	100	99.48 +2.91	0	3.74 +1.74	0	0	<1 +1.69	<1 +1.41	0	<1 +1.41		
DDA/4A2/78/C2006	65	46.87 +2.52	0	7.92 +1.69	35	30.04 +1.80	<1 +1.41	<1 +1.41	0	<1 +1.41		
DDA/4A3/78/C2006	79	52.05 +2.61	0	16.34 +1.65	21	15.16 +1.49	<1 +1.27	<1 +1.27	0	<1 +1.27		
DDA/4X2/78/C2006	48	N/A	52	N/A	0	N/A	N/A	N/A	0	N/A		
DDA/4X3/78/C2006	70	41.72 +2.24	30	16.56 +1.49	0	6.02 +1.38	<1 +1.14	<1 +1.14	0	<1 +1.14		
DDA/4X /78/C2006	79	75.07 +3.15	9	11.47 +1.67	0	<1 +1.75	11.77 +1.18	11.77 +1.18	12	11.77 +1.18		
DDA/JP-8/78/C2006	0	9.76 +2.45	0	<1 +1.78	0	<1 +1.60	<1 +1.42	<1 +1.42	0	<1 +1.42		
DDA/8A /78/C2006	0	3.41 +2.54	0	<1 +1.79	31	38.92 +2.12	<1 +1.24	<1 +1.24	0	<1 +1.24		
DDA/8A3/78/C2006	0	10.43 +2.44	0	<1 +1.77	15	20.77 +2.45	<1 +1.23	<1 +1.23	0	<1 +1.23		
DDA/8X2/78/C2006	0	20.99 +2.36	48	33.39 +1.70	0	3.41 +1.55	<1 +1.27	<1 +1.27	0	<1 +1.27		
DDA/8X3/78/C2006	0	15.12 +2.40	24	19.88 +1.64	0	7.20 +1.50	<1 +1.27	<1 +1.27	0	<1 +1.27		
DDA/8CM/78/C2006	0	18.24 +2.38	0	17.61 +1.65	0	<1 +1.60	6.64 +1.84	6.64 +1.84	12	6.64 +1.84		

cited in the fuels containing 2040 solvent in Table 7 from the proposed values are well beyond the confidence limits set on the predicted values. In particular, retained samples DDA/4A2/78/C2006, DDA/8A2/78/C2006, and DDA/8A3/78/C2006 all have 2040 about 8-10 volume percent higher than initially proposed.

As for the ignition fuels set (Table 8), all samples analyzed had both JP-4 and JP-8 present. Consequently, the predicted component concentrations are inherently more accurate for this set, since the original calibration curves were geared for complex mixtures containing both JP-4 and JP-8.

## SECTION IV

### CONCLUSIONS

1. The determination of selected distillate blending solvents in thoroughly complex aircraft fuel matrices can easily be accomplished via glass capillary gas chromatography.
2. The accuracy of the analyses, as exemplified by back-calculations, is remarkable since the methodology was so simple, avoiding the inherently more accurate but more tedious internal standardization techniques.
3. The method is relatively fast, requiring only about 70 minutes between sample injections.
4. JP-4 and any of three blending solvents could be determined volumetrically in JP-8 with a precision of  $\pm 1-4\%$  absolute at the 95% confidence level.
5. The method is completely automated except for the derivation of the line equations and the precision statements, but sophisticated laboratory automation systems could accommodate all data reduction easily.

SECTION V

FUTURE WORK

1. If the need arises for a concerted support effort, shorter capillary columns could provide sufficient resolution to reproduce this analytical method with the same or better precision and accuracy but at substantially reduced analyses times.
2. Again, if necessary, more accurate determinations are possible with an as yet uninvestigated internal standardization technique.
3. A laboratory data automation system, to be requisitioned for this laboratory, will be utilized to save considerable method development and data reduction time for similar projects in the future.

APPENDIX

# Monsanto

**MONSANTO RESEARCH CORPORATION**

Dayton Laboratory  
1515 Nicholas Road  
Dayton, Ohio 45407  
Phone (513) 268-3411  
TWX 810-459-1681

26 November 1979

Mr. Paul Hayes, Jr.  
Wright Patterson Air Force Base

SUBJECT: PREDICTING THE UNCERTAINTY IN A VALUE OF X (VOLUME PERCENT OF A GIVEN COMPONENT) ASSOCIATED WITH AN OBSERVATION OR SERIES OF OBSERVATIONS, ON THE DEPENDENT VARIABLE Y (AREA PERCENTAGES AS DETERMINED BY THE GAS CHROMATOGRAPHIC INTEGRATOR).

Using the output from your subroutine STATIST, and using the equations on pp. 231-232 of "Statistical Analysis in Chemistry and the Chemical Industry" by C. A. Bennet and N. L. Franklin, J. Wiley & Sons, 1954; we need the following parameters:

b = the slope of the original calibration curve

$\sigma^2 =$  square of the standard error of estimate

$Y_o =$  the mean of the m new observations, (where m = # of new observations)

$\bar{y} =$  the mean of the original area percentages used in developing the calibration curve.

$X_i =$  the original values of volume percentages used to derive the calibration curve

n = the number of points (pairs) in the original calibration curve

t-value = value of student's t from Table 1. (with m-1 degrees of freedom)

Taking the JP-4 data as an example, we have (from the computer printout):

b = 0.37481

$\sigma^2 =$  (Standard error of estimate)<sup>2</sup> = (0.71524)<sup>2</sup>

$\sigma^2 =$  0.51157

.....Con't.

Mr. Paul Hayes  
Wright Patterson Air Force Base  
26 November 1979

$$n = 37$$

Taking the points at  $X = 40$ , for illustration only, we compute:

$$Y_o = (14.43 + 13.85 + 13.59 + 13.96)/4 \text{ (at } X_o = 40)$$

$$Y_o = 13.96 \text{ (with } m = 4)$$

$$\bar{y} = 9.48$$

Using equation (g) on page 232:

$$\text{Var}(X_o) \sim \frac{\sigma^2}{b^2} \left[ \left( \frac{1}{m} + \frac{1}{n} \right) + \frac{(Y_o - \bar{y})^2}{b^2 \sum (X_i - \bar{X})^2} \right] \quad (1)$$

We make use of the following identity:

$$\sum (X_i - \bar{X})^2 = \sum (X_i)^2 - \frac{(\sum X_i)^2}{n}$$

In this case:

$$\sum (x_i - \bar{X})^2 = 11158$$

and

$$(Y_o - \bar{y})^2 = 20.07$$

Substituting into equation (1) we have:

$$\text{Var}(X_o) \simeq \frac{0.51157}{0.14048} \left[ \left( \frac{1}{4} + \frac{1}{37} \right) + \frac{20.07}{(0.14048)(11158)} \right]$$

$$\text{Var}(X_o) \simeq (3.6416) [(0.27703) + (0.012804)]$$

$$\text{Var}(X_o) \simeq 1.0554$$

$$S(X_o) = \text{Var}(X_o)^{1/2}$$

$$S(X_o) \simeq 1.0273$$

The 100  $(1 - \alpha)\%$  confidence limits for  $X_o$  are:

.....Continued

Mr. Paul Hayes, Jr.  
Wright Patterson Air Force Base  
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Page 3

$$X_0 - t(n-2, 1 - \frac{\alpha}{2}) S(X_0) < \bar{x} + \frac{y_0 - \bar{y}}{\beta} < X_0 + t(n-2, 1 - \frac{\alpha}{2}) S(X_0) \quad (2)$$

This equation simply states that the 95% confidence interval for the true value of  $X_0$

where the true value of  $X_0$  is:

$$\text{True value of } X_0 = \bar{x} + \frac{y_0 - \bar{y}}{\beta}$$

and  $\beta$  = true slope

lies between  $X_0 + t(n - 2, 1 - \alpha/2) S(X_0)$

and  $X_0 - t(n - 2, 1 - \alpha/2) S(X_0)$

If we select  $\alpha = 0.05$  (i.e. the 100 (1 - 0.05)% or 95% confidence level) then from Table 1

$$t(37 - 2), 1 - \frac{0.05}{2} = t(35, 0.025) = 2.344$$

$$(40 - 2.411) < \text{True value of } X_0 < (40 + 2.411)$$

Sincerely,

*Ed Eimutis*

E. C. Eimutis,  
Contract Manager

ECE:lh

Attachment: Table 1

CC: F. N. Hodgson

TABLE 1. PERCENTAGE POINTS OF THE t-DISTRIBUTION\*

$\alpha$	0.50	0.25	0.10	0.05	0.025	0.01	0.005
1	1.00000	2.4142	6.3138	12.706	25.452	63.657	127.32
2	0.81650	1.6036	2.9200	4.3027	6.2053	9.9248	14.089
3	0.76489	1.4226	2.3534	3.1825	4.1765	5.8409	7.4533
4	0.74070	1.3444	2.1318	2.7764	3.4954	4.6041	5.5976
5	0.72769	1.3009	2.0150	2.5706	3.1634	4.0321	4.7733
6	0.71756	1.2733	1.9432	2.4469	2.9687	3.7074	4.3168
7	0.71114	1.2543	1.8946	2.3646	2.8412	3.4995	4.0293
8	0.70639	1.2403	1.8595	2.3060	2.7515	3.3554	3.8325
9	0.70272	1.2297	1.8331	2.2622	2.6850	3.2498	3.6897
10	0.69981	1.2213	1.8125	2.2281	2.6338	3.1693	3.5814
11	0.69745	1.2145	1.7959	2.2010	2.5931	3.1058	3.4966
12	0.69548	1.2089	1.7823	2.1788	2.5600	3.0545	3.4284
13	0.69384	1.2041	1.7709	2.1604	2.5326	3.0123	3.3725
14	0.69242	1.2001	1.7613	2.1448	2.5096	2.9768	3.3257
15	0.69120	1.1967	1.7530	2.1315	2.4899	2.9467	3.2860
16	0.69017	1.1937	1.7459	2.1199	2.4729	2.9208	3.2520
17	0.68919	1.1910	1.7396	2.1098	2.4581	2.8982	3.2225
18	0.68837	1.1887	1.7341	2.1009	2.4450	2.8784	3.1966
19	0.68763	1.1866	1.7291	2.0930	2.4334	2.8609	3.1737
20	0.68696	1.1848	1.7247	2.0860	2.4231	2.8453	3.1534
21	0.68635	1.1831	1.7207	2.0796	2.4138	2.8314	3.1352
22	0.68580	1.1816	1.7171	2.0739	2.4055	2.8188	3.1188
23	0.68531	1.1802	1.7139	2.0687	2.3979	2.8073	3.1040
24	0.68485	1.1789	1.7109	2.0639	2.3910	2.7969	3.0905
25	0.68443	1.1777	1.7081	2.0595	2.3846	2.7874	3.0782
26	0.68405	1.1766	1.7056	2.0555	2.3788	2.7787	3.0669
27	0.68370	1.1757	1.7033	2.0518	2.3734	2.7707	3.0565
28	0.68335	1.1748	1.7011	2.0484	2.3685	2.7633	3.0469
29	0.68304	1.1739	1.6991	2.0452	2.3638	2.7564	3.0380
30	0.68276	1.1731	1.6973	2.0423	2.3596	2.7500	3.0298
40	0.68066	1.1653	1.6839	2.0211	2.3289	2.7045	2.9712
60	0.67862	1.1616	1.6737	2.0003	2.2991	2.6603	2.9146
120	0.67656	1.1559	1.6577	1.9799	2.2699	2.6174	2.8599
$\infty$	0.67449	1.1503	1.6449	1.9600	2.2414	2.5758	2.8070

\* Computed by Maxine Merrington from "Tables of Percentage Points of the Incomplete Beta Function," *Biometrika*, 32 (1941), pp. 168-181, by Catherine M. Thompson, and reproduced by permission of Professor E. S. Pearson.

A description of this table is given in Section 4.54. Where necessary, interpolation should be carried out using the reciprocals of the degrees of freedom. The function 120 is convenient for this purpose.

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